

Using Optically Stimulated Electron Emission as an Inspection Method to Monitor Surface Contamination

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INTRODUCTION

During redesign of the Space Shuttle reusable solid rocket motor (RSRM), NASA amended the contract with ATK Launch Systems (then Morton Thiokol Inc.) with Change Order 966 to implement a contamination control and cleanliness verification method. The change order required:

- A quantitative inspection method
- A written record of actual contamination levels versus a known reject level
- A method that is more sensitive than existing methods of visual and black light inspection

Black light inspection is only useful for inspection of contaminants that fluoresce near the 365 nm spectral line and is not useful for inspection of most silicones that will not produce strong fluorescence. Black light inspection conducted by a qualified inspector under controlled light is capable of detecting Conoco HD-2 grease in gross amounts and is very subjective due to operator sensitivity.

Optically stimulated electron emission (OSEE), developed at the Materials and Process Laboratory at Marshall Space Flight Center (MSFC), was selected to satisfy Change Order 966.

OSEE offers several important advantages over existing laboratory methods with similar sensitivity, e.g., spectroscopy and nonvolatile residue sampling, which provide turn around time, real time capability, and full coverage inspection capability. Laboratory methods require sample gathering and in-lab analysis, which sometimes takes several days to get results. This is not practical in a production environment. In addition, these methods do not offer full coverage inspection of the large components.

OSEE THEORY OF OPERATION

The OSEE method utilizes the photo-electric effect (Figure 1). The OSEE sensor contains an ultraviolet (UV) bulb which, when directed onto a surface, causes electrons to be emitted from the surface. The freed electrons are attracted to a positively charged collector ring in the sensor that is attached to a direct current (DC) battery. The resulting electron current is displayed on the instrument readout and collected by a data acquisition system. If a contaminant exists on the surface, the UV radiation that reaches the steel is reduced, which in turn reduces the current.

CORRELATION OF OSEE TO CONTAMINATION

Since the implementation of OSEE, a number of correlation studies have been performed to establish quantitative OSEE values. The correlation studies were conducted using Conoco HD-2 grease as the contaminant to establish the minimum OSEE values. HD-2 grease is applied on RSRM hardware as a preservative. Other studies have been performed using silicone products, which demonstrated that OSEE is also sensitive to silicone contamination.

Proper correlation studies require well controlled laboratory protocol, i.e., carefully followed procedures and repeatable methods of contamination application involving nominal amounts of contamination at various levels to establish an OSEE curve. Typical levels used in the correlation studies were 5, 10, 20, and 30 milligrams of grease per square foot. The procedure used to apply the contaminant was developed at MSFC by ATK personnel and utilizes a modified ultrasonic printed circuit board solder flux spraying machine. The contaminant is mixed in a solvent solution and is delivered to the test sample by the sprayer in a controlled amount using a syringe pump. During application, the solution is atomized and blown onto the panels by an air knife. The panels are moved across the path of the air knife at a controlled speed. Contamination levels are measured using pre-weighed foils that are processed at the same time as each panel. Contamination levels are

controlled with syringe pump pressure, speed through the sprayer, energy levels applied to the ultrasonic atomizing knife, and the ratio of grease to solvent. After the panels are prepared and the contamination levels are verified to meet the nominal levels desired, the panels are inspected with OSEE using a noncontact instrument. The result is OSEE values that can be correlated with known contamination levels.

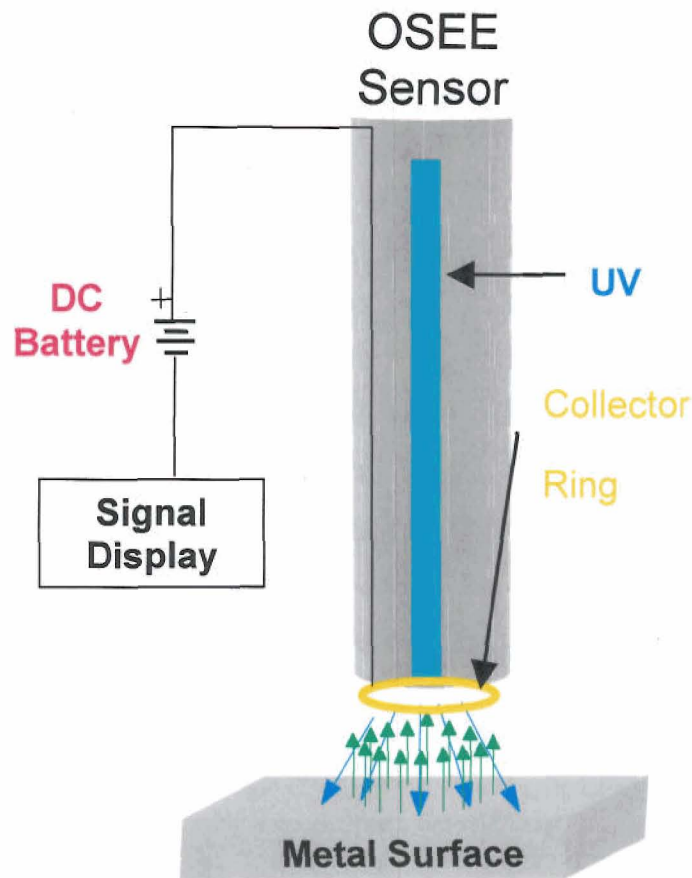


Figure 1: OSEE Theory of Operation

BASELINE AND CALIBRATION OF OSEE

The OSEE instrument used at MSFC during the early testing phase became the starting point for baseline tracking. The baseline OSEE values were determined by using the instrument to inspect a surface chemistry stable chromium standard (chrome vapor deposited on glass). Due to the fragile nature of the chromium standard, stainless steel calibration transfer standards were used to transfer the baseline value to steel for on-line calibration at ATK Launch Systems. A standard instrument was set up and calibrated to the MSFC baseline and was then maintained in a clean room to reduce the risk of instrumentation drift. For production calibration, a clean transfer standard was inspected with the control instrument, a value was derived from the inspection for that standard, and the printout of that value was attached to the canister that contained the panel. The standard was placed in a sealed canister and taken to the production line. When an operator was ready to perform an on-line inspection, he took a reading on the panel and adjusted the electrical gain on the line instrument to obtain the value on the printout attached to the standard. A "properly cleaned transfer standard" was one that underwent a tightly controlled cleaning process using laboratory grade solvents and cleaners. Properly cleaned transfer standards had an acceptable OSEE value that was tracked in a database. Transfer standards, as well as verification with several backup control instruments, constituted the process to ensure that the baseline for calibration did not drift.

A new instrument, which included bulb current adjustment capability, offered the first opportunity for electronic calibration. With electronic calibration, the UV bulb energy is measured and adjusted to a defined set point for each

instrument and compensation is made for bulb differences and aging. Electronic calibration includes connecting a 100 gig-ohm resistor to the OSEE collector which simulates the signal current. The gain is then adjusted to a defined set point. The premise is that if several instruments are set up to the defined set points for bulb energy and amplifier gain, they will produce like outputs when measuring a test sample. Initially, this ATK developed calibration method did not work; however, research conducted at the NASA Langley Research Center brought about a solution. The mercury arc lamp used in the OSEE instrument emits spectrum at 254 nm, 185 nm, and several spectral lines between 297 nm and 313 nm. The 254 nm spectral line emits 95 percent of the energy from the mercury arc lamp and in theory much of the 185 nm energy is absorbed by air, so it had been assumed that the OSEE signal was due to the 254 nm spectral line. The research at NASA Langley demonstrated that the 185 nm spectral line is the spectral line that produces the OSEE signal. A successful electronic calibration procedure was performed and implemented into production in 1994. Implementation of the ATK developed electronic calibration made it possible to switch from the transfer plate system to tracking baseline by tracking UV bulb energy values, instrument gain values, and calibration auditing.

Electronic calibration and the subsequent improvements to the instrument have improved reliability, repeatability, and traceability to the National Institute of Standards and Technology. It is important to note that some of the energy at the 185 nm spectral line is absorbed by oxygen and therefore the signal-to-noise ratio for this method is not optimum. This continues to be an issue but does not invalidate the calibration method. Verification of the calibration functionality has been achieved weekly over the past eight years by successful audits of the calibration.

CALIBRATION AUDITING

All OSEE instruments used in production are audited weekly. The instruments are set up side-by-side and calibrated to nominal values. Each instrument is used to inspect the same set of test panels. All instruments are required to be within a set tolerance relative to each other. The audit is also performed quarterly on MSFC OSEE instruments. This audit ensures that the calibration is working properly and that all the instruments are still tracking each other. It is a secondary method to maintain baseline.

OSEE IN THE PRODUCTION ENVIRONMENT

Spot check inspections are performed in many process locations within the production areas. The spot checks are performed to a sampling plan that is fixed within the data acquisition software (Figure 2). Following instrument calibration, the operator places a 0.250-in. standoff on the sensor and inspects the part (Figure 3). ATK has continued to make improvements to the instrumentation, such as the extended sensor in Figure 3, and data acquisition software upgrades as computer and operating systems have evolved.

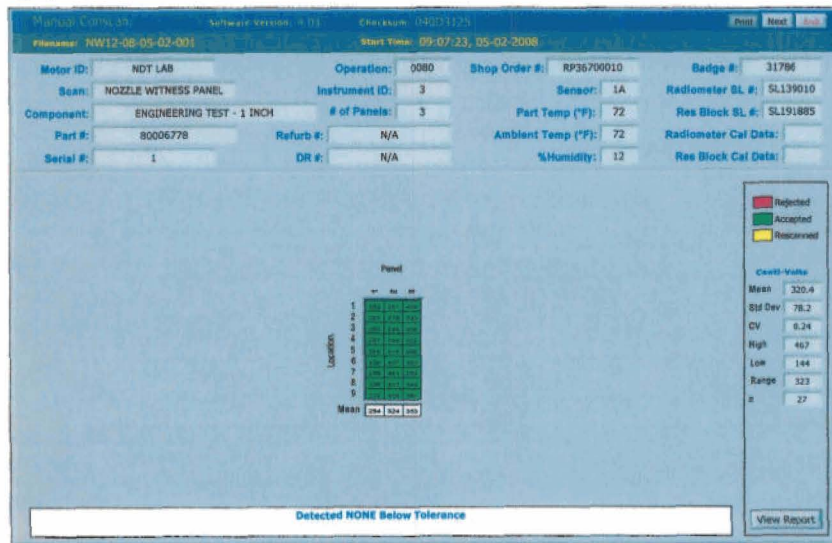


Figure 2: Manual Software Display

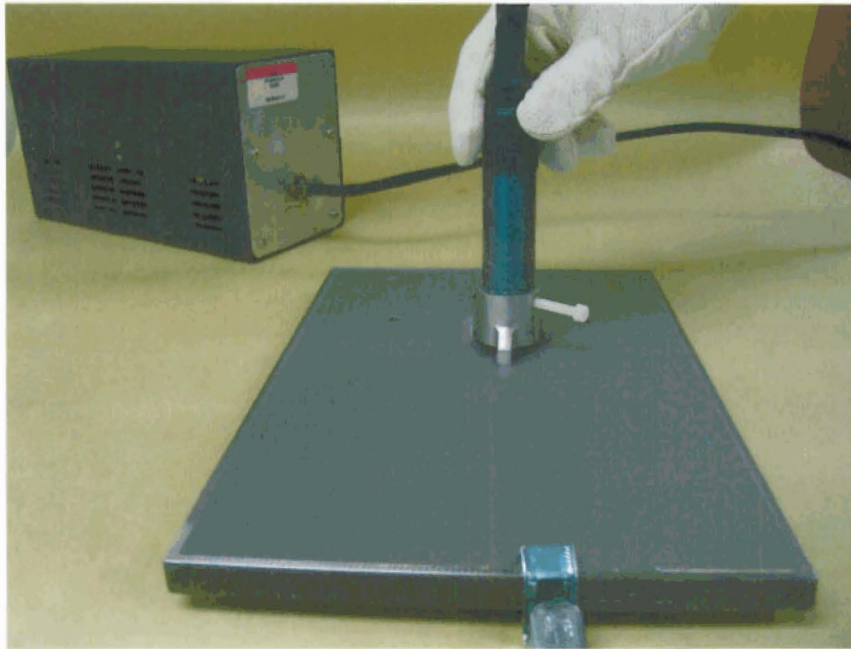


Figure 3: Manual Conscan Inspection Sensor

About ninety percent of the 1,000 square foot area of the RSRM case hardware is inspected in an automated process just prior to adhesive bonding operations. The OSEE sensor is mounted to a robotic arm that maintains the 0.250-in. standoff using an eddy current sensor and closed loop motorized controller. As the case rotates on a turn table, case run out and wobble occurs. The eddy current sensor provides an error signal to the controller that moves the OSEE sensor to compensate for the standoff error. The robotic arm is mounted to a paint mechanism that runs up or down as the case rotates in a barber pole-like scan. Location data from the turn table and paint mechanism, as well as the OSEE signal, are fed into a computer. A resultant mapping of the location and OSEE signal is displayed (Figure 4) on the data acquisition display. The various colors correlate to the amount of contamination on the case.

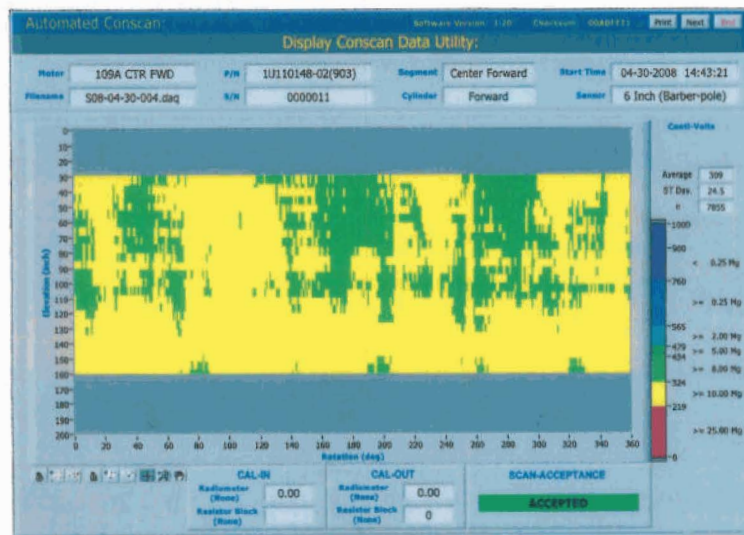


Figure 4: Automated Software Display

ISSUES WITH OSEE IN THE PRODUCTION ENVIRONMENT

The OSEE instrument is very sensitive to contamination, but is also sensitive to oxidation. Two weeks can pass between grit blast cleaning and the final automated OSEE inspection of the RSRM case hardware. The resultant oxidation reduces the OSEE signal and will produce what may be termed as a false negative in the OSEE signal. During production, it is always assumed that if the OSEE signal is below the minimum requirement it could be contaminated and therefore cleaning is required even though it may also be possible that the low reading is due to oxidation. Testing at MSFC has included aged steel correlation tests which have lowered the minimum acceptable OSEE values to compensate for the oxidation issue. A portable Fourier transform infrared (FTIR) spectrometer is being tested at MSFC that will allow for onsite verification of low value OSEE signals without the lengthy delay of laboratory methods. This will reduce or eliminate what may be unnecessary hand cleaning. Due to the signal-to-noise issue with the 185 nm spectral line, the OSEE signal is affected by air born particulates, solvent fumes, and other factors not yet fully understood. To compensate for these factors, dry times are required after using solvents and prior to OSEE inspections. Basic overall housekeeping in the production areas are performed which helps to control air born particulates. Solvents have become a larger issue in recent years with the introduction of the new non-ozone depleting chemicals (ODC) that are now used in production. Further testing is being performed to better understand what effects these non-ODC cleaners have on the OSEE signal.

OSEE AS A PROCESS MONITOR

Many years of production use and testing at MSFC have shown that the OSEE instrument is extremely sensitive to production variation. This variation could be due to differences in grit blast angles, grit blast standoff, the delay time between grit blast and inspection, changes in the cleaning solvents, and just about any other process variation. In production areas that have very tightly controlled processes, the OSEE signal is very predictable and consistent, in production areas that have uncontrollable variation, the OSEE signal is much less predictable. Experience with the OSEE instrument has shown that the more controlled the process, the less variation there is in the OSEE data.

CONCLUSION

ATK has been successful in the implementation and use of the OSEE instrument not only for contamination detection, but also for production process monitoring. OSEE implementation and use has not been without issues and concerns, many of which are still being addressed.

REFERENCES

1. Gauss, Raymond L., , "A Noncontacting Scanning Photoelectron Emission Technique for Bonding Surface Cleanliness Inspection", *NASA TM-100361*, 1989
2. Huddleston, Odell Jr., Mattes, Robert A., "Baseline Photoemission Study of D6AC Steel", *Thiokol TWR-63543*, 1992
3. Huddleston, Odell Jr., Mattes, Robert A., Cauthen, Otis, "OSEE (Conscan) Response to Conoco HD-2 Grease Contamination In D6AC Steel, *Thiokol TWR-64901*, 1993
4. Huddleston, Odell Jr., Mattes, Robert A., Cauthen, Otis, "The Effect of Grit Blast Angle On OSEE and the EA913NA/D6AC Steel Bond System", *Thiokol TWR-64900*, 1993
5. Huddleston, Odell Jr., "Precision Contamination Application Using Ultrasonic Spray Technology" *Thiokol TWR-65882*, 1994
6. Huddleston, Odell Jr., Mattes, Robert A., "Electronic Calibration Method for OSEE (Conscan) Instrumentation", *Thiokol TWR-65884*, 1994
7. Huddleston, Odell Jr., Mattes, Robert A., "OSEE Correlation to Contamination on D6AC Steel and 7075-T73 Aluminum" *Thiokol TWR-65883*, 1995
8. Mattes, Robert A., "Contamination Monitoring of RSRM Bonding Surfaces using OSEE", 1989
9. Plowman, Ronald M., "Conscan and Bonding Evaluation for Case Processing", *Thiokol TWR-65244*
10. Smith, Tennyson, "Quantitative Techniques for Monitoring Surface Contamination", *Surface Contamination-Genesis, Detection, and Control*, Plenum Press, New York, 1979
11. Welch, Christopher S., Abedin, M. Nurul, Yost, William T., "Optically Stimulated Electron Emission: Current-Voltage Response and Spectral Sensitivity", *NASI-18347*, College of William and Mary, NASA Langley Research Center, Plenum Press New York, 1992

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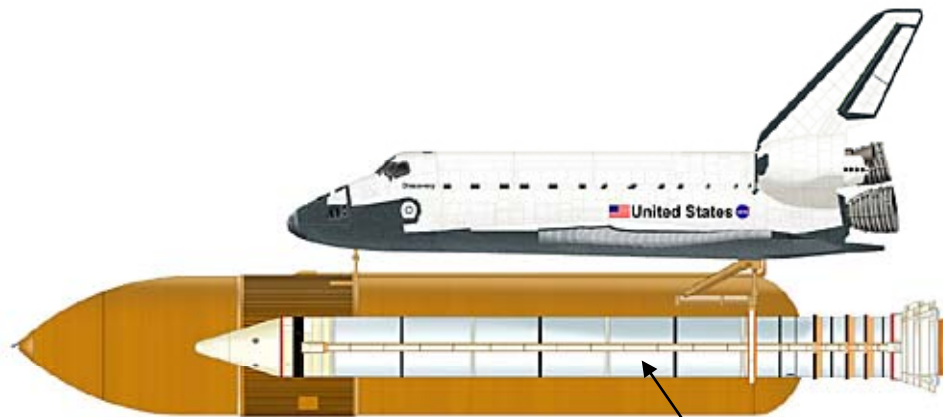


Reusable Solid Rocket Motor



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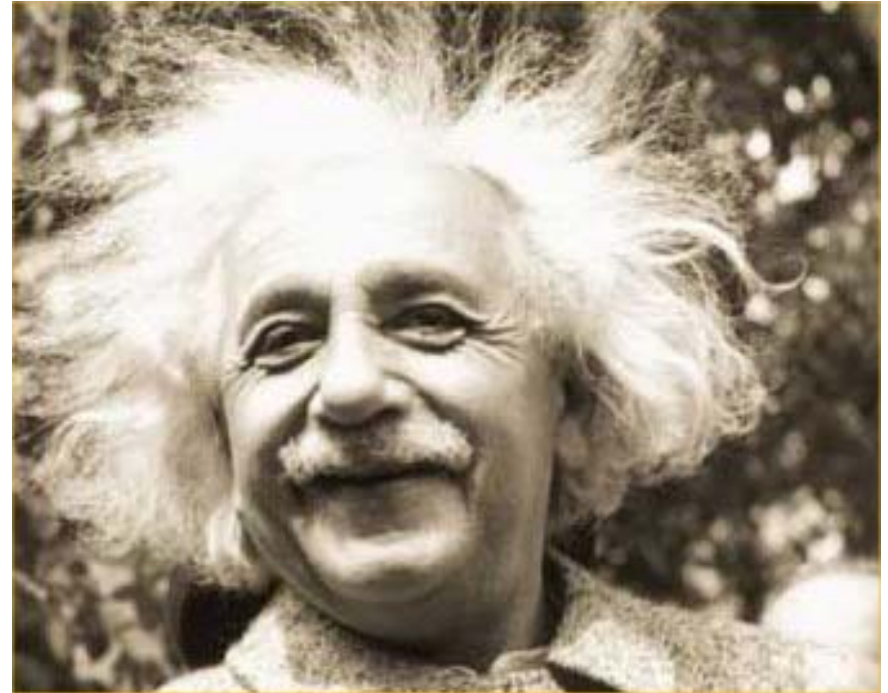
Space Transportation System



Reusable Solid Rocket Motor (RSRM)

- During redesign of the reusable solid rocket motor (RSRM), NASA amended the contract with ATK (then Morton Thiokol) to include a method to inspect for contamination
 - The amendment required
 - A quantitative inspection method
 - A written record of actual contamination levels versus a known reject level
 - A method that is more sensitive than existing methods of visual and black light inspection
 - Optically stimulated electron emission (OSEE), developed at Marshall Space Flight Center (MSFC) for RSRM application, was selected as the method to satisfy the contract amendment

- Albert Einstein's Theory of Relativity made him famous, but it was his explanation of the photoelectric effect and its contribution to Quantum Theory that won him the Nobel Prize in 1921
- OSSE utilizes the photoelectric effect

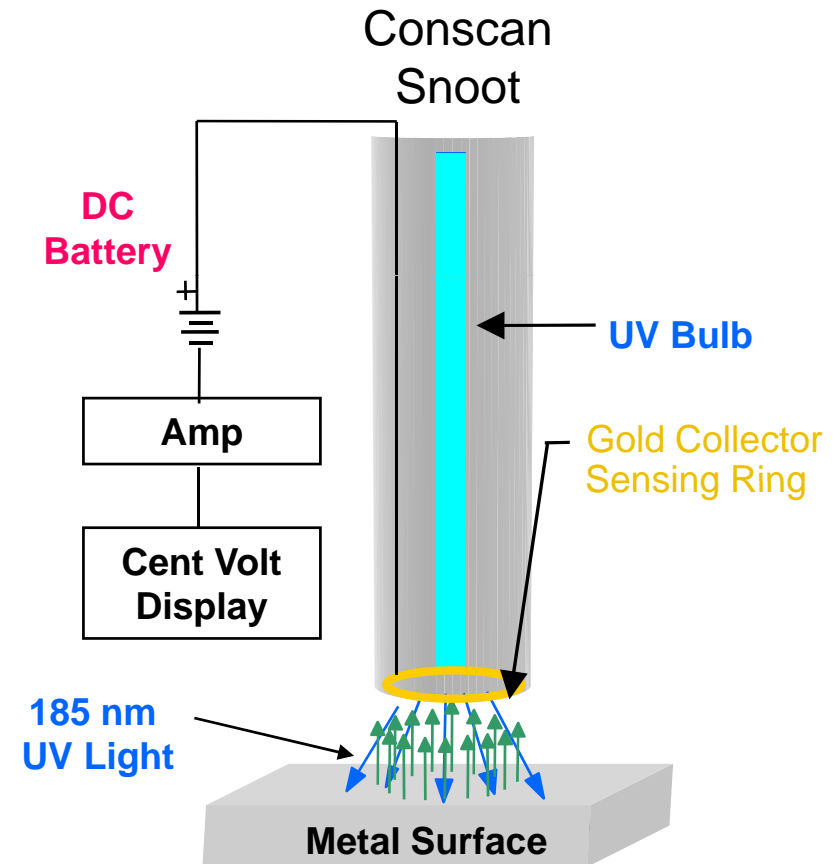


OSEE Theory of Operation

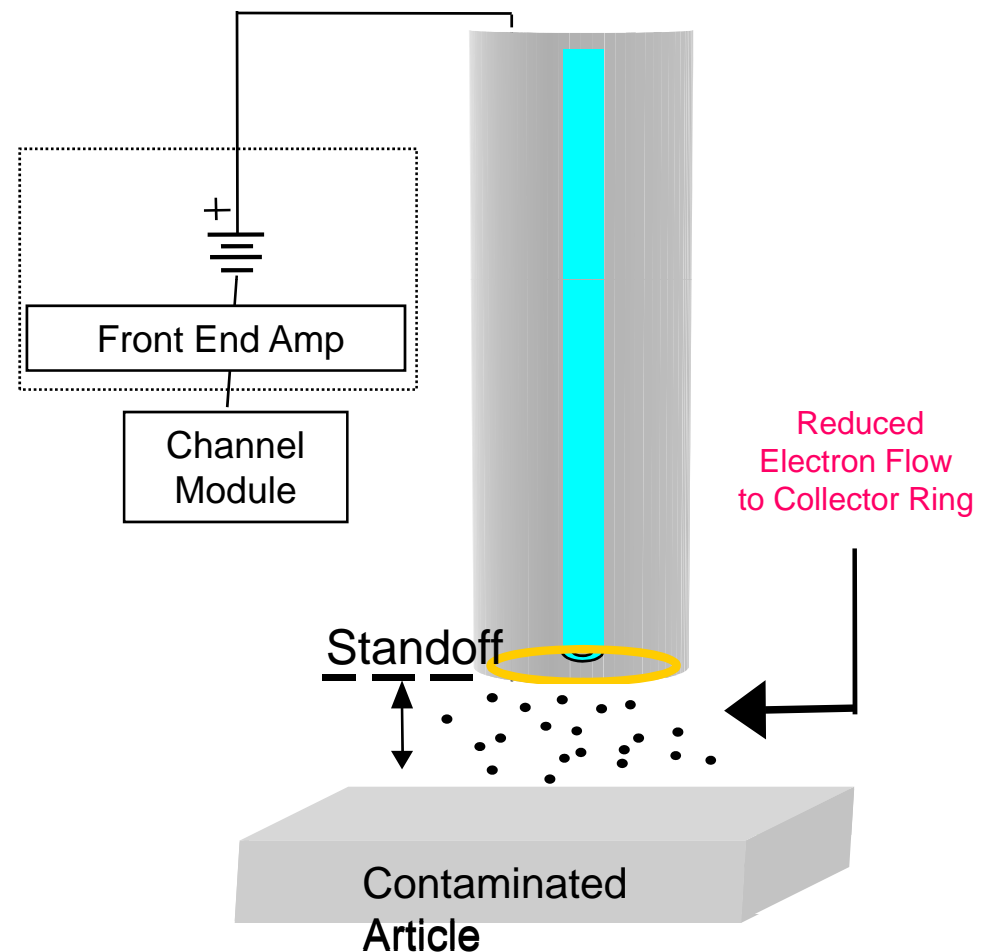


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- D6AC steel emits electrons from the surface when irradiated with UV light at the 185 nm wavelength
- Increasing or decreasing the UV intensity increases or decreases the number of electrons emitted
- The electrons emitted from the surface are collected on a charged ring at the end of the OSEE sensor, amplified, and displayed as a voltage



- The distance from the sensor to the substrate surface (standoff) is held constant at 0.250 inch
- Contamination on the surface of the substrate reduces the electron flow to the sensor, resulting in a lower value



- Correlation studies were conducted using Conoco HD-2 grease as the contaminant to establish the minimum OSEE values
- Typical levels of grease measuring 5, 10, 20, and 30 milligrams of grease per square foot were used to develop the correlation
- Grease was mixed in a solvent solution and delivered to the test sample using an off-the-shelf modified solder flux machine built by Sonotek
 - The solution is atomized and blown onto the panels by an air knife
 - The panels are moved across the path of the air knife at a controlled speed
 - Contamination levels are measured using pre-weighed foils that are processed at the same time as each panel
 - Panels are inspected with a non-contacting OSEE instrument
 - The result is OSEE values that can be correlated with known contamination levels
 - Bond strength studies have also performed with the contaminated panels

OSEE Correlation to Contamination



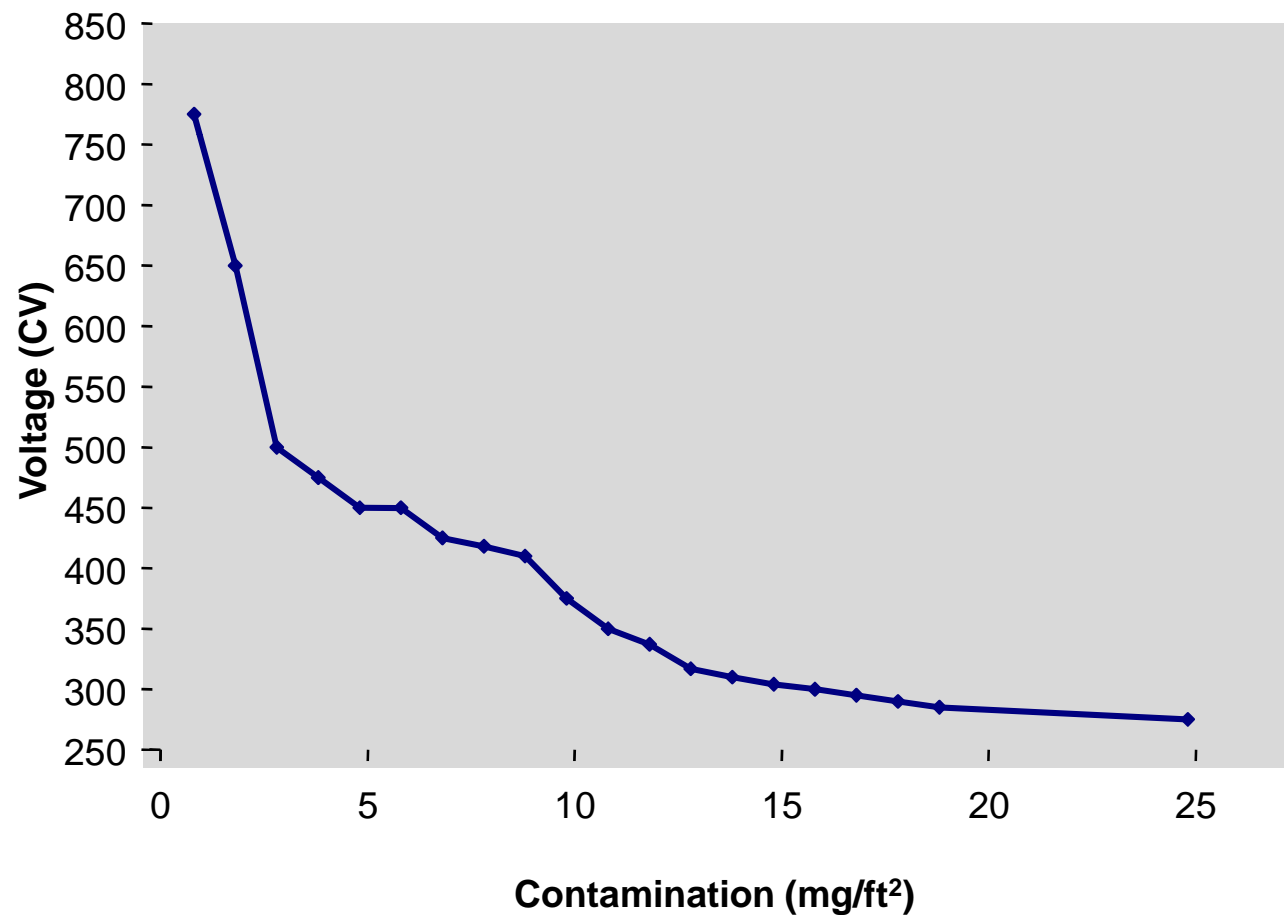
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Sonotek Solder Flux Machine



OSEE Voltage Versus Surface Contamination



- The premise for the electronic calibration method is that if basic functional parameters such as UV lamp energy and instrument gain are set to a defined set point, then instruments will provide like data when measurements are taken
- Early attempts with the calibration method were unsuccessful because all efforts attempted to calibrate using the 254 nm spectrum
- It was thought that the 254 nm spectrum caused the photoelectric effect on D6AC steel during inspections at ATK
 - The mercury vapor lamp used in the OSEE instrument emits UV spectrum at 254 nm and 185 nm with the 254 nm producing the majority of the energy
 - It is understood that much of the energy at the 185 nm spectrum is absorbed by air
- Research at NASA Langley demonstrated that the 185 nm spectral line is the spectral line that produces the OSEE signal on D6AC steel
- Electronic calibration has improved reliability and repeatability, and provided traceability to the National Institute of Standards and Technology

OSEE Electronic Calibration

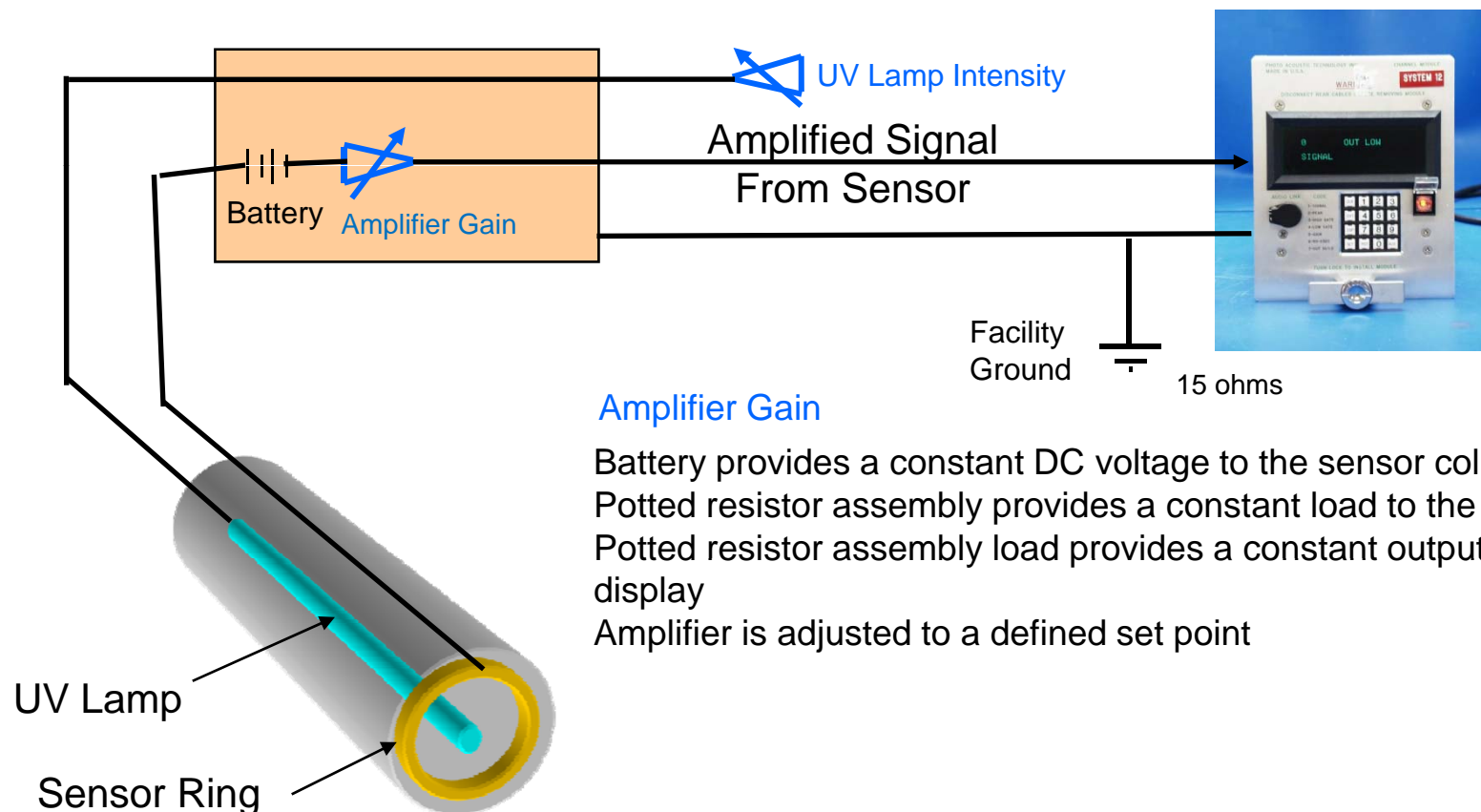


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UV Lamp Intensity

Bulb output is measured at the 185 nm wavelength

Bulb current is adjusted for a defined radiometer reading



Amplifier Gain

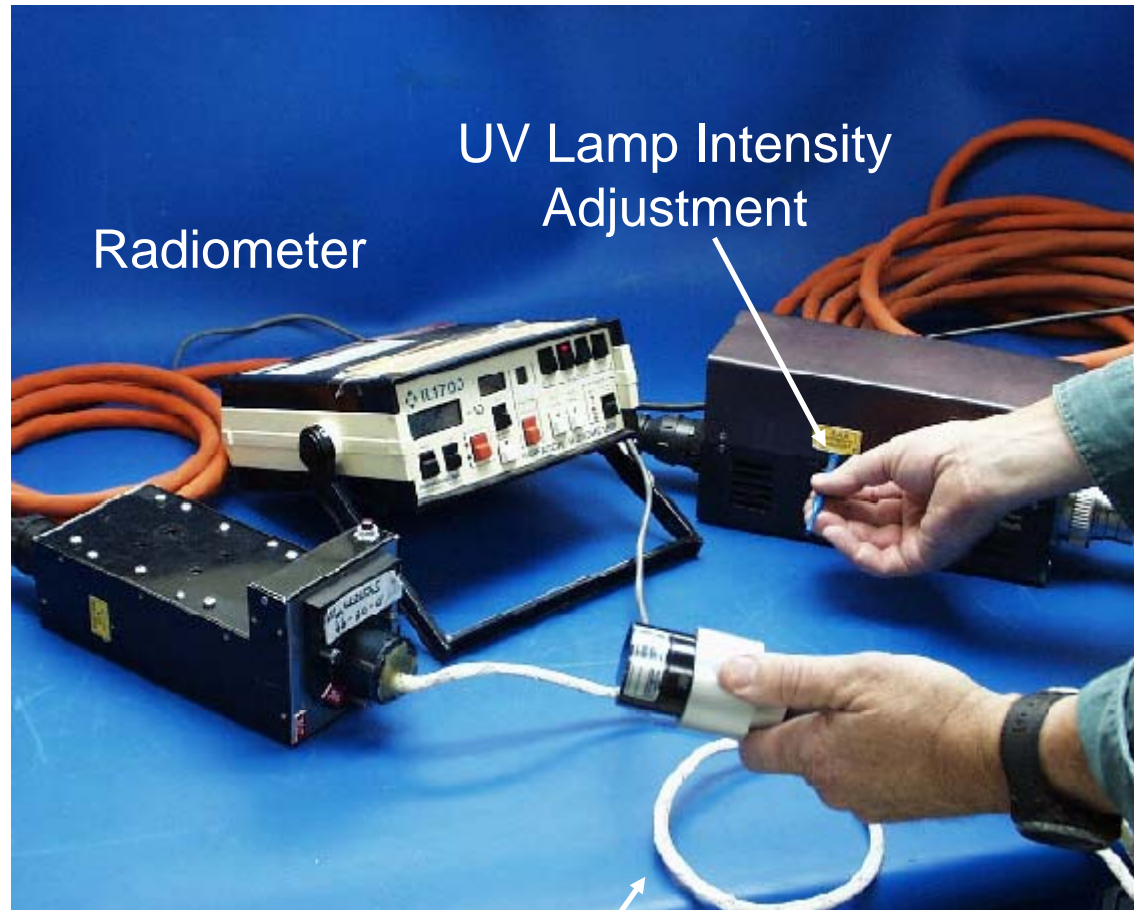
Battery provides a constant DC voltage to the sensor collector
Potted resistor assembly provides a constant load to the battery
Potted resistor assembly load provides a constant output to the display
Amplifier is adjusted to a defined set point



OSEE Electronic Calibration



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- Due to the fact that the 185 nm spectrum is a small energy level and is absorbed in air, the calibration is not optimum
- An audit of the calibration was established to monitor the process
 - Instruments in the audit are electronically calibrated
 - Instruments are used to inspect the same 3 witness panels in controlled locations
 - Data from the inspection must be less than 10 percent variation
- Careful control of the UV lamps and instrumentation has produced successful audits for 8 years

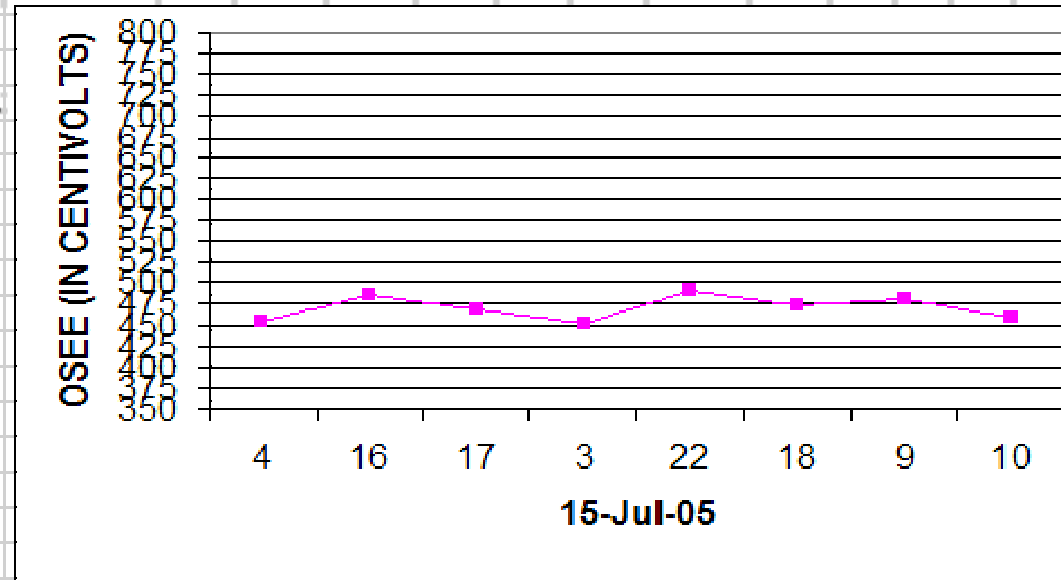
OSEE Electronic Calibration Audit



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ID	Plate	Plate	Plate	Aug	Balls	PS	PS	25mm	Balls	Hum	Auto	In Hg	Batt	Disp	Calc	Stat	AD
	CF	CF	CF	CF	Install	mm	mm	output	F	%	F		Volt		Nr		Delta
	38									38	69	30.02					
4	476	435	454	455	1-Mar-05	17.2		4.69	144				72.7	2	11	688	-1
16	686	466	487	486	12-May-05	17.4		4.86	146				73.4	2	3	686	-1
17	494	444	487	488	25-Apr-05	22.3		3.35	153				89.8	8	1	588	8
3	468	438	455	451	1-Feb-05	14.7		4.83	134				78.3	1	1	559	-1
22	511	472	498	491		17.1		4.1	147				78.6	4	5	588	-1
18	483	461	476	473		16.6		4.74	138				74.1	8	8	618	8
9	582	458	486	482	19-Sep-02	16.9		3.65	158				67.4	4	18	533	8
18	475	452	453	488	31-Jan-01	14.9		4.98	134				78.4	8	8	888	8

Grand Average				471
Overall %	8.8%	9.3%	7.9%	8.6%

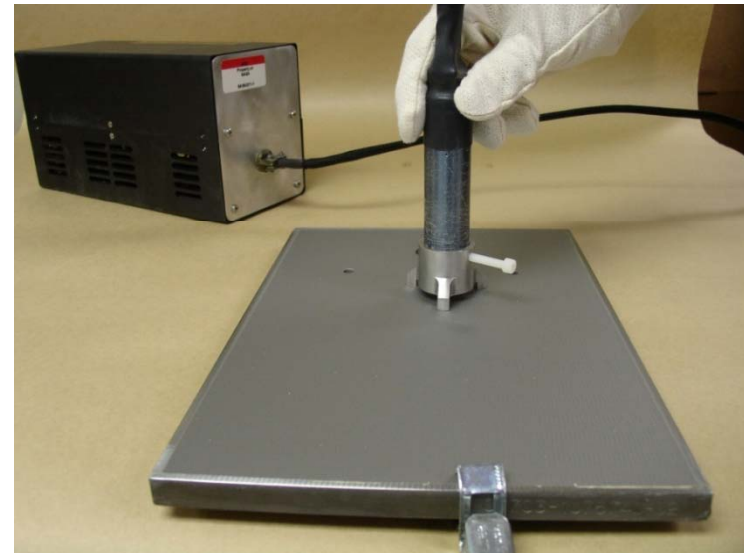


- OSEE is used as an in-process spot check for many RSRM components
- The spot checks are performed to a predetermined sampling plan and are controlled with software in the data acquisition system
- A small hand-held sensor is used for the spot checks
- The software retains the data for the inspection record

OSEE in Production



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Manual Conscan: Software Version: 4.03 Checksum: 040D3125

Filename: NW12-08-05-02-001 Start Time: 09:07:23, 05-02-2008

Motor ID: NDT LAB Operation: 0080 Shop Order #: RP36700010 Badge #: 31786

Scan: NOZZLE WITNESS PANEL Instrument ID: 3 Sensor: 1A Radiometer SL #: SL139010

Component: ENGINEERING TEST - 1 INCH # of Panels: 3 Part Temp (°F): 72 Res Block SL #: SL191885

Part #: 80006778 Refurb #: N/A Ambient Temp (°F): 72 Radiometer Cal Data:

Serial #: 1 DR #: N/A %Humidity: 12 Res Block Cal Data:

Panel		Centi-Volts		
Location	Panel	1	2	3
1	352	251	434	
2	281	278	345	
3	240	248	388	
4	297	144	323	
5	298	315	288	
6	290	467	357	
7	299	401	283	
8	298	401	443	
9	299	400	383	
Mean	284	324	383	

Detected NONE Below Tolerance

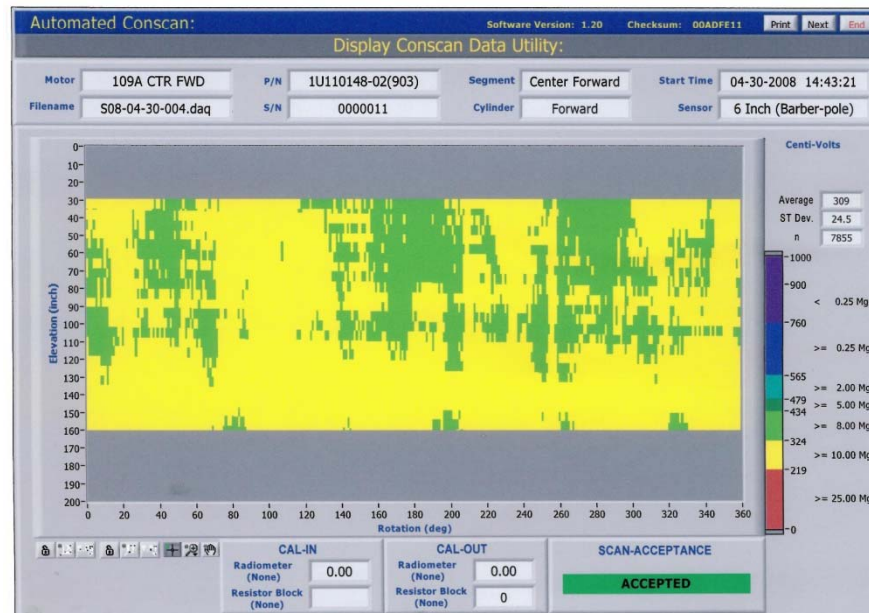
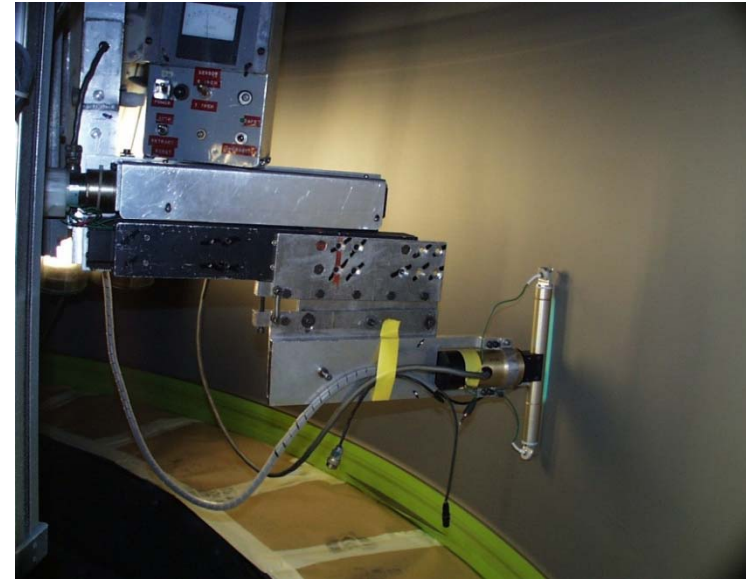
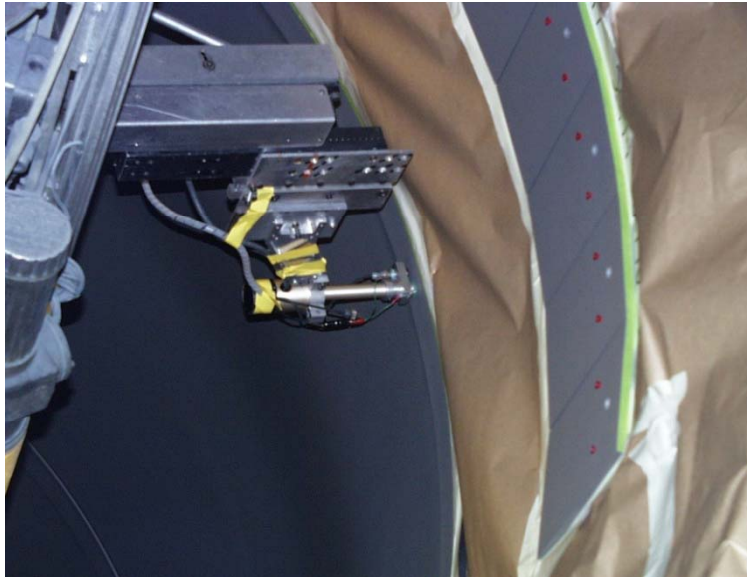
View Report

- Approximately 90 percent of each RSRM case assembly is inspected using automated OSEE prior to bond operations
- The sensor is mounted on a robotic arm that maintains a 0.250-inch standoff using an eddy current sensor and closed-loop motorized controller
- A mast allows longitudinal translation of the sensor and the case assembly rotates on a turntable
- Location data from the turntable and longitudinal translation , as well as the OSEE signal, are fed into the data acquisition computer
- The result is a mapping of color-coded OSEE values versus inspection locations that are displayed and retained as part of the inspection record

OSEE in Production



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Issues with OSEE in the Production Environment



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- The OSEE instrument is very sensitive to contamination, but is also sensitive to oxidation
- Many weeks can pass between grit blast cleaning and the final automated OSEE inspection of the RSRM case hardware
- The resultant oxidation reduces the OSEE signal and produces what may be termed as a false negative in the OSEE signal
- The oxidation issue has helped drive production to improve time lines between grit blast and bonding
- Testing at MSFC has included aged steel correlation tests, which have lowered the minimum acceptable OSEE values to compensate for the oxidation issue
- Several portable Fourier transform infrared spectrometers have recently been introduced in the market and are being evaluated
- This will allow for onsite verification of low value OSEE signals without the lengthy delay of laboratory methods

Issues with OSEE in the Production Environment



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- Because of the low energy of the 185 nm spectrum, there is a lot of sensitivity to air born particulates, solvent fumes, and other factors not fully tested
- To compensate for these factors, dry times are required after using solvents and prior to OSEE inspections
- Basic overall housekeeping in the production areas is performed, which helps control air born particulates
- Solvents have become a larger issue in recent years with the introduction of the new non-ozone depleting cleaners (ODC) now used in production
- Further testing is being performed to better understand what effects these ODCs have on the OSEE signal

Issues with OSEE in the Production Environment



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- Many years of production use and testing at MSFC have shown that the OSEE instrument is extremely sensitive to production variation
- This variation could be due to differences in grit blast angles, grit blast standoff, the delay time between grit blast and inspection, and changes in the cleaning solvents
- In production areas that have tightly controlled processes, the OSEE signal is predictable and consistent
- In production areas that have less control over variation, the OSEE signal is much less predictable with more variation
- Experience with the OSEE instrument has shown that the more controlled the process, the less variation there is in the OSEE data

- ATK has successfully implemented and used the OSEE instrument not only for contamination detection, but also for production process monitoring
- OSEE implementation and use is not without issues and concerns, many of which are still being addressed

Questions?